

Pilot of the Pollution Prevention Technology Application Analysis Template

Utilizing

PolyIonix Polymer Filtration Technology

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DISCLAIMER

This document pilots the Pollution Prevention (P2) Technology Application Analysis Template (P2 template) on the PolyIonix Separation Technologies, Inc. Polymer Filtration (PF) technology. It is designed to assist the user in analyzing the application of P2 technologies. While it provides a template for the general types of questions that you should ask when evaluating a P2 technology, it may not include all of the questions that are relevant to your business, or which your business is legally required to ask.

This document is not an official U.S. EPA or Army Corps of Engineers guidance document and should not be relied upon as a method to identify or comply with local, state or federal laws and regulations. EPA and the Army Corps of Engineers have not examined, nor do they or their contractor endorse, any technology analyzed using the P2 template.



Introduction

The purpose of this technology application analysis is to illustrate how the Pollution Prevention (P2) Technology Application Analysis Template (P2 template) can summarize a technology, in this case the PolyIonix Polymer Filtration (PF) Technology, an innovative P2 technology which has been utilized in a commercial application at Silvex Surface Technology (Silvex) in Westbrook, ME. The purpose of this technology application analysis is twofold: first, to assist users of P2 technologies in evaluating the applicability of this technology to their needs; second, to assist vendors of P2 technologies in developing their own technology application analyses.

The technology application analysis characterizes, in a concise manner, the main features of the technology, its benefits, the costs associated with its implementation, regulatory aspects, and lessons learned from the application experience. Additional useful information beyond that included in this technology application analysis is available from PolyIonix.

The intent of the Environmental Protection Agency (EPA) in developing and piloting the P2 template is to promote the use of technology application analyses as a method of promoting and accelerating the introduction and use of new P2 technologies.

PolyIonix was selected as the General Industrial Partner to Los Alamos National Laboratory (LANL) to commercialize the PF technology. This innovative technology, winner of a R&D 100 Award in 1995, was developed at LANL. PolyIonix is currently developing PF systems for site-specific applications. Currently, standardized units are produced for the metal-finishing industry, but the potential exists for the use of the PF technology in other industrial applications.

This technology application analysis presents the implementation of the newly-patented PF technology field trial at Silvex to demonstrate the removal and recovery of nickel ions from rinse water generated in the electroplating process at the facility. The field trial ran for approximately three weeks and processed 90 gallons of nickel rinse water per day, at concentration levels of 100-250 ppm. Information regarding batch process performance, hull cell testing to characterize the electroplate, and polymer washing with permeate water was obtained. Additionally, the quality of the PF permeate discharge water was compared to the Silvex process water (city water).

This technology application analysis is divided into seven sections:

- Introduction
- Description of P2 Technology
- P2 Technology Application
- P2 Technology Performance
- Cost Information
- Regulatory / Safety Requirements
- Lessons Learned / Implementation Issues



Description of P2 Technology

In electroplating, or depositing a protective coating of metal on an object, the item to be treated is cleaned and then passed through an electroplating bath of metal ions in solution and then washed in a series of rinsing baths. Typically, when the process is complete, the electroplating metals that remain in the rinse water are precipitated, collected, and buried as toxic sludge. The resulting sludge contains valuable materials, but when disposed is a potential environmental hazard.

The PolyIonix PF technology is a system to recover and recycle metal ions from metal-finishing process streams. This technology provides a cost-effective means to recover and reuse metal ions in electroplating operations, and reduce metal ion concentrations in effluent discharge to levels which meet or are below regulatory discharge criteria.

The primary goals of the PF technology are to **MINIMIZE** electroplating wastes through selective hazardous metal-ion removal from process streams; **REALIZE** energy and economic savings, and environmental benefits from the implementation of new technology; and **RECYCLE** valuable resources.

The following section describes the PolyIonix PF technology, giving information on the major equipment, feed influent and product effluent characterization, and energy / utility requirements. The applicability of this technology to industry is also described. In addition, the advantages and limitations in applying this technology are provided.

Technology Description

Overview

The PF technology selectively removes metal ions, such as nickel and zinc, from contaminated waste water. The PF technology combines metal-binding, water-soluble chelating polymers (which form a complex by joining the polymer to the metal ion) with advanced ultrafiltration membranes, as presented below in Figure 1.

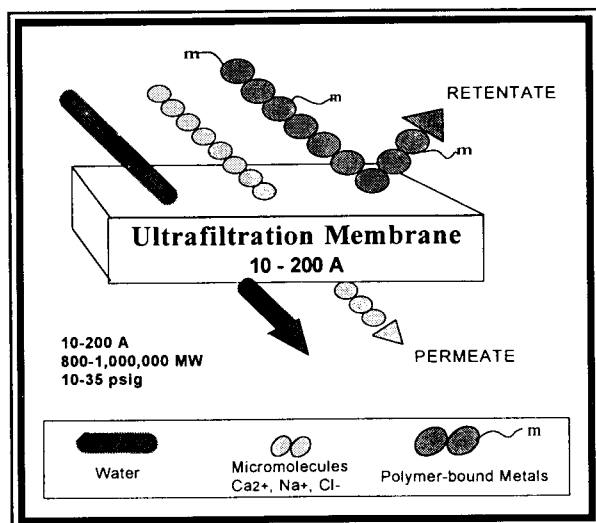


FIGURE 1 - SEPARATION DIAGRAM

SEPARATION IS BASED ON SIZE EXCLUSION

RETENTATE:

Polymer/Metal Complex is Physically Too Large to Pass Through the Ultrafiltration Membrane

PERMEATE:

Unbound Species Readily Pass Through the Ultrafiltration Membrane



The PF technology performs the following three functions on metal-bearing waste water streams:

- **BINDING** - Binding of the metal ions in the waste water is achieved through polymer addition. Selective binding of target metals with unique polymers temporarily increases the effective size of the polymer/metal complex, and the ions are filtered from the water.
- **CONCENTRATION** - Concentration can be accomplished by off-the-shelf ultrafiltration membranes.
- **RECOVERY** - In the metal recovery phase, the metals are released from the polymer via pH adjustment. Polymer, metal, and water are reclaimed.

The PF technology can be run in either batch or semi-continuous mode, as specified by the customer. During the batch process, the volume of the solution in the tank decreases linearly with time. In addition, the concentration of metal increases hyperbolically with time. During the semi-continuous process, the volume in the process tank remains constant.

The semi-continuous process continuously feeds the rinse solution at the same rate as the water permeates from the membrane. In the semi-continuous mode, a typical day's volume is concentrated and processed throughout the day. After that, the hold up volume in the process tank continues to be processed as if in batch mode.

Either process mode allows the reuse of water and polymer, and recycles the metal back into the plating process. Upon regeneration, the polymer is put back into the holding tank and can process rinse water in the system again. Figure 2 presents a closed-loop electroplating process and illustrates where the PF process takes the rinse water and processes it to reclaim metal and water for process use.

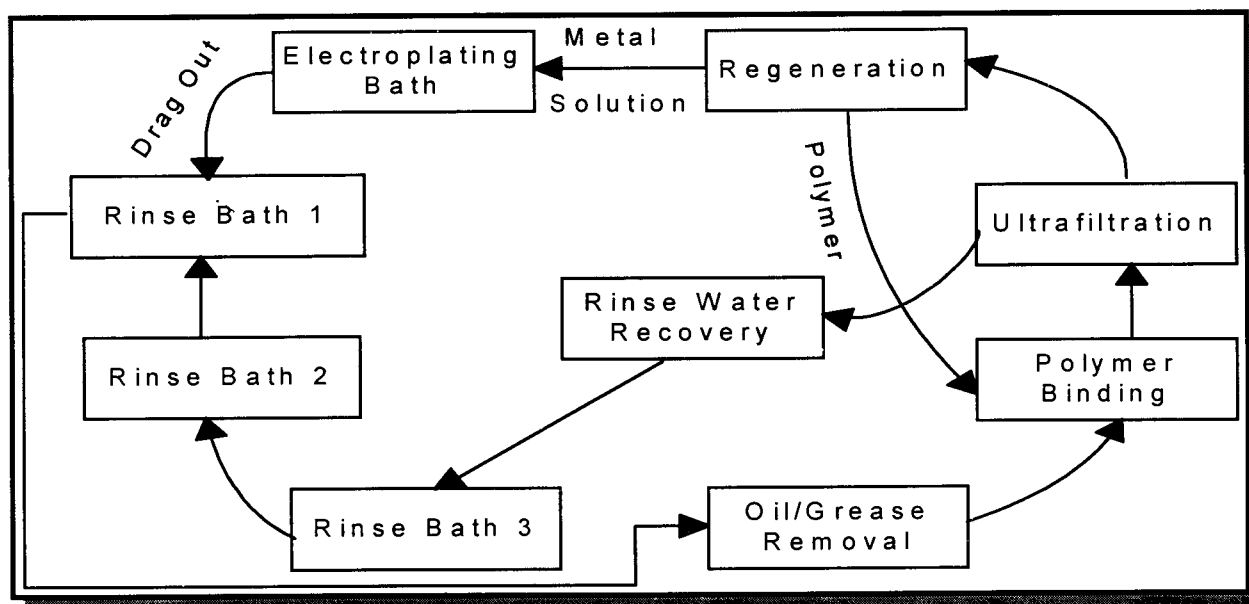


FIGURE 2 - CLOSED-LOOP ELECTROPLATING PROCESS DIAGRAM

While the PF technology has been demonstrated at relatively low volumes and flow rates, scale-up to high flow rates to tens of thousands of gallons per day is both possible and economically feasible, according to the vendor. Each application site has different circumstances. Scale-up

costs are dependent on how the PF technology fits in to the existing industrial process, and the degree of automation required for the PF system, as well as other site-specific variables.

Patents have recently been awarded for the process and polymer compositions. Improved manufacturing processes have both reduced the cost and improved the quality and technical capability of off-the-shelf membranes used in the PF system.

Detailed Description

A schematic of the generic PF process is presented below in Figure 3. As shown in Figure 3, metal-bearing waste water (influent) is treated in the reaction reservoir, where the polymer binds with the metal ions under balanced acid/base conditions (pH control). The reservoir fluid is then pumped through the ultrafiltration system, a cartridge packed with ultrafiltration membranes shaped into hollow fibers. As the fluid flows across the membrane, water and other small molecules (simple salts such as calcium and sodium) pass through the porous membrane walls of the fibers and are discharged through the outlet (permeate reservoir). The polymer-bound metal is too large to pass through the pores, and becomes concentrated inside the hollow fibers. This material is then returned (retentate recycle) to the fluid reservoir. In this manner, 500 gallons of rinse water can be treated and the metal ions concentrated to approximately 15-20 gallons in 4-6 hours in a typical 1 to 1.5 gpm plating system.

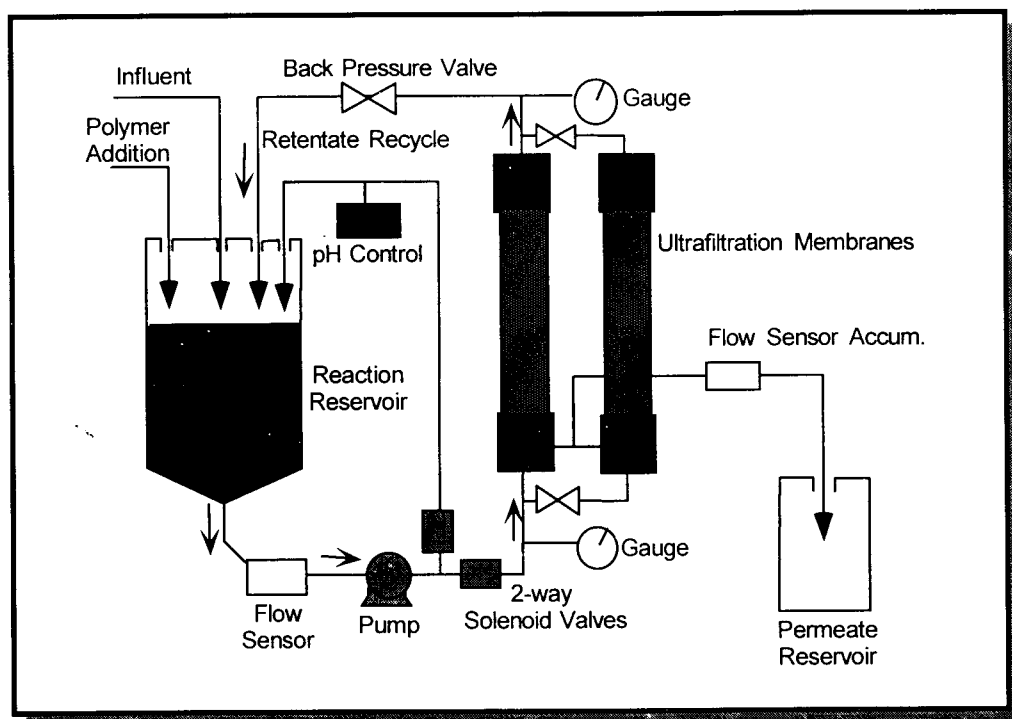


FIGURE 3 - SCHEMATIC OF PF PROCESS

During the metal recovery stage of the process, the pH of this concentrate is adjusted to break the metal/polymer complex. The concentrate is then diafiltered in continuous mode to wash out free metal ions. On average, more than 95% of the metals that entered the system during the concentration phase are reclaimed during diafiltration, or metal recovery. The final solution containing the reclaimed metal ions can be recycled back into the electroplating process. Thus, the overall volume reduction achieved in this treatment process is approximately 4:1. The cleaned

polymer chelant is reused in the next binding/concentration process.

PolyIonix has designed and constructed a PF system for Silvex to remove nickel salts from rinse water of nickel plating operations. The self-contained PF system used at Silvex is highly mobile, and is pictured below. The Silvex system contains all the necessary components to accept, process, and reclaim metal rinse water. The footprint of the unit is approximately 6 ft x 3 ft.

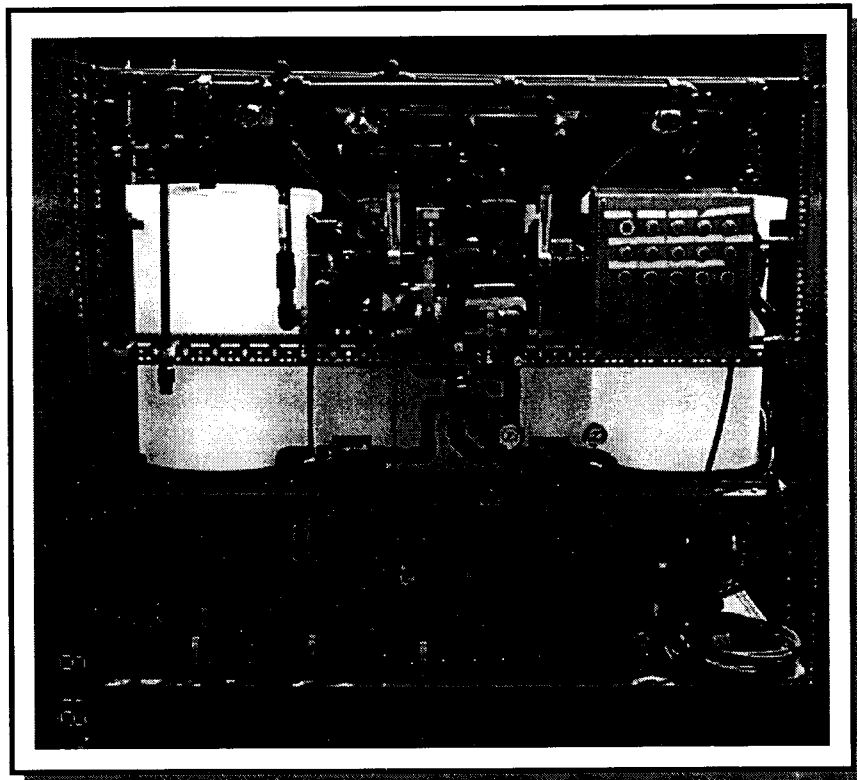


FIGURE 4 - 1-1.5 GPM PF UNIT USED AT SILVEX

PF SYSTEM COMPONENTS:

- ➡ **Water Soluble, Metal Binding Polymer** which is added to the unit's 30 gallon reaction reservoir on the left side of the cart.
 - ➡ **Horizontal Ultrafiltration Membrane Cartridge** on the upper tier of the piping.
 - ➡ **Controls** for monitoring the recovery process can be seen on the front control panel.
- | | |
|--|--------------------------------|
| - Static Mixer | - 30 Gallon Reaction Reservoir |
| - 5 Micron Filter and 1 Micron Filters | - 30 Gallon Permeate Reservoir |
| - Acid and Base Tanks | - Pumps, Plumbing |

Technology Applicability

This section describes the applicability of this technology to users, the development history, and the advantages/limitations claimed by the technology vendor.

Applicability to Industry/User

The primary focus of the PF technology commercialization efforts has been directed towards the Electroplating/Metal Finishing industry, primarily due to the following:

- metal ions used in plating operations (Department of Energy, Department of Defense, and commercial) are currently discharged for burial as waste hydroxide sludge;
- hydroxide sludge does not meet current waste stabilization criteria and is a potential environmental hazard;
- valuable metals are being wasted by disposal of this sludge;
- the number of licensed disposal sites is decreasing, and disposal costs are rising; and
- hazardous waste is an environmental liability.

The PF technology development initially targeted nickel recovery from electroplating rinses. However, this technology may be applied to other transition metals, such as zinc and copper. Future developments for this technology will be broadened to include other applications such as precious metals and other metal contaminants such as mercury and lead. With the ability to remove other metals, the PF technology will apply to other industrial applications, as shown below.

OTHER APPLICATIONS

- Mining
- Acid mine drainage
- Municipal waste waters
- Pulp and Paper
- Soil remediation
- Battery manufacturing
- Electronics waste water
- Textile waste water



Development / Application History

Table 1 shows the developmental summary of the PolyIonix PF technology:

TABLE 1 - APPLICATION HISTORY OF PF TECHNOLOGY

Time	Location	Scale	Target Metals	Capacity	Length of Run	Reason for Termination
1995	Boeing Aerospace, Seattle, WA	Demonstration of Technology	Nickel, Zinc	2-5 gallons per minute	2 weeks	Met performance criteria
June 1996	State Plating, Elwood, IN	First Commercial Demonstration	Nickel (Chloride)	500 gallons per day	4 weeks	Met performance criteria
June 1997	Theta Plate, Albuquerque, NM	Second Commercial Demonstration	Nickel (Chloride, Sulfate)	300 gallons per day	4 weeks	Met performance criteria
October 1997	Silvex Surface Technology, Westbrook, ME	Third Commercial Demonstration	Nickel (Chloride, Sulfate)	150 gallons per day	3 weeks	Met performance criteria

Lessons Learned During P2 Technology Development**Lessons Learned**

The following are lessons learned during development of the technology which have already been incorporated into the design of the standard unit:

- ▶ Automation of PF technology (valves, pumps, etc.) is required to reduce labor intensity of operation.
- ▶ Adequate pre-filtration to remove particles > 5 um is required.
- ▶ Final permeate is of sufficient quality that it can be reused within the process.
- ▶ The PF technology demonstrates that metal removal (nickel) to levels ranging from 0.1-2.0 ppm, is easily achieved, and is below typical regulatory discharge limits.



The following are lessons learned during development of the technology which need to be accounted for in tailoring the unit to an application setting:

- ▶ Proper waste stream characterization is mandatory. It is necessary to know the characteristics of the metal-bearing waste stream to be treated so that application of the appropriate polymer is determined and the applicability of the PF technology to the existing process is assessed.
- ▶ Purified metal ions (nickel) can be returned to the plating bath for reuse without loss of plating performance.
- ▶ Based on preliminary engineering designs and layout drawings, the system can accommodate increases in size and flow rate (up to at least 10,000 gallons per day) by adding ultrafiltration membranes, larger pipe diameters, and possibly making other mechanical adjustments.
- ▶ Thorough data gathering is essential. This includes, but is not limited to, definition of appropriate data quality objectives, and execution of sampling and testing, process and performance analysis, and economic data gathering.

According to PolyIonix, some lessons learned resulted in definition of the primary advantages resulting from the PF technology application:

ADVANTAGES

- **METAL ION RECYCLABILITY:** Recovery of pure nickel ion concentrate for recycling and reuse.
- **FAST REACTION BINDING:** Metal ions do not have to cross organic phase or liquid/solid boundaries to reach binding sites.
- **LOADING CAPACITY:** Because they have more accessible binding sites, the PF technology's polymers have loading capacities 3-8 times greater than ion-exchange resins.
- **SELECTIVITY:** The polymers can be tailored to have specific binding sites for specific metal ions, binding sites that reject benign impurities such as calcium potassium, and other salts.
- **INSTANTANEOUS KINETICS:** Minimal residence time in the process tanks.
- **NO SLUDGE FORMATION:** Near zero discharge.
- **LOW ENERGY:** 2 electric motors and low pressure requirements: input pressure of less than 30 psi, and a pressure differential across the membrane of greater than 10 psi.
- **LOW CAPITAL COSTS:** Approximately \$45,000 for a typical system with 1 to 1.5 gpm capacity. (Includes Reaction Reservoir, Pumps, Plumbing, Controls, Chelating Polymer, Ultrafiltration Membrane Cartridges, Acid and Base Tanks)
- **REUSE:** Maximizes use of valuable metals without extensive recovery or refining efforts, and allows for reuse of water and polymer.



P2 Technology Application

The following Section describes the use of the PolyIonix PF technology at Silvex Surface Technology in Westbrook, ME. This section further describes the details of the PolyIonix PF technology location within the Silvex plant and how the plant production/operation was affected.

P2 Technology Application

General Setting

A PF technology for removing nickel salts from rinse water of nickel plating operations was designed and operated at Silvex, an electroplating job shop located in Westbrook, ME.

Silvex has a number of different electroplating lines, three of which plate nickel. The demonstration was associated with two of three nickel plating lines at Silvex. Figure 5 below presents the flow of product through the two nickel lines at this electroplating facility which were used in the demonstration. The third nickel line was not included because it was not operational during the time of the PolyIonix demonstration at Silvex. Therefore, it is not shown on Figure 5.

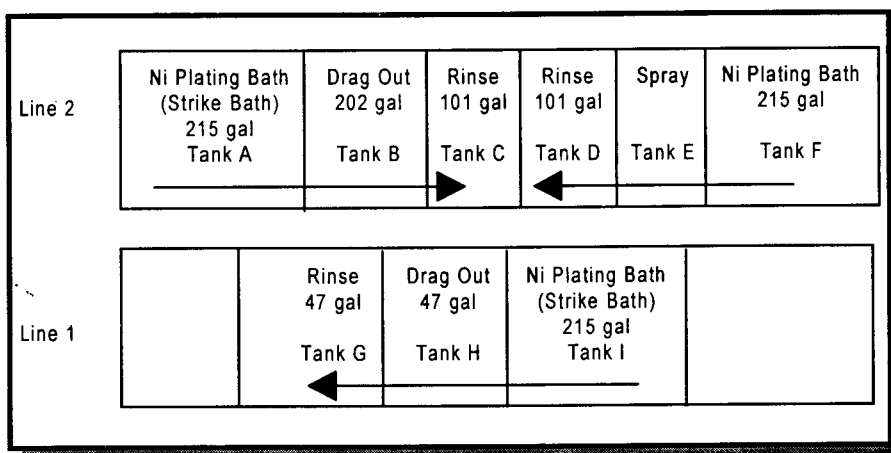


FIGURE 5 - SILVEX NICKEL PROCESS DIAGRAM (PARTIAL)

The PF technology received rinse water from several nickel rinse tanks, including Tanks C, D, and G in Figure 5 above. For the Silvex application, rinse water from the rinse tanks was transferred to a common 250 gallon holding tank. The average volume of the rinse solution treated was approximately 90 gallons per day at a rate of 1.5 gpm.

Polymer is added to the nickel rinse water from the holding tank to form a chelate solution. The chelate solution is pumped through an ultrafiltration membrane which retains the metal/polymer complex and permeated water containing trace amounts (much less than the regulatory discharge limit) of nickel salts. At Silvex, a typical process batch is 90 gallons of nickel rinse water containing approximately 200 ppm nickel. At the end of the final ultrafiltration phase, the batch is



concentrated to approximately 7.5 gallons with a nickel concentration of 4500 ppm. The PF unit was designed to operate at a flow rate of 1.5 gpm on nickel rinse water from the electroplating process. Figure 6 presents the Treatment Process Flow Diagram for the 1.5 gpm nickel rinse water process at Silvex.

Technology Implementation

The PF technology consists of a 30 gallon process tank to which the nickel rinse solution is fed and polymer is added, and in which pH adjustment is performed by acid/base addition. The chelate solution is pumped through a static mixer and through the ultrafiltration membrane in both the concentration and metal recovery phases. Inlet and outlet pressures of the chelate solution are measured across the ultrafiltration membrane. The system incorporates a reverse flow design, which minimizes the need for membrane cleaning. Figure 6 presents the process flow diagram for the concentration and metal recovery processes.

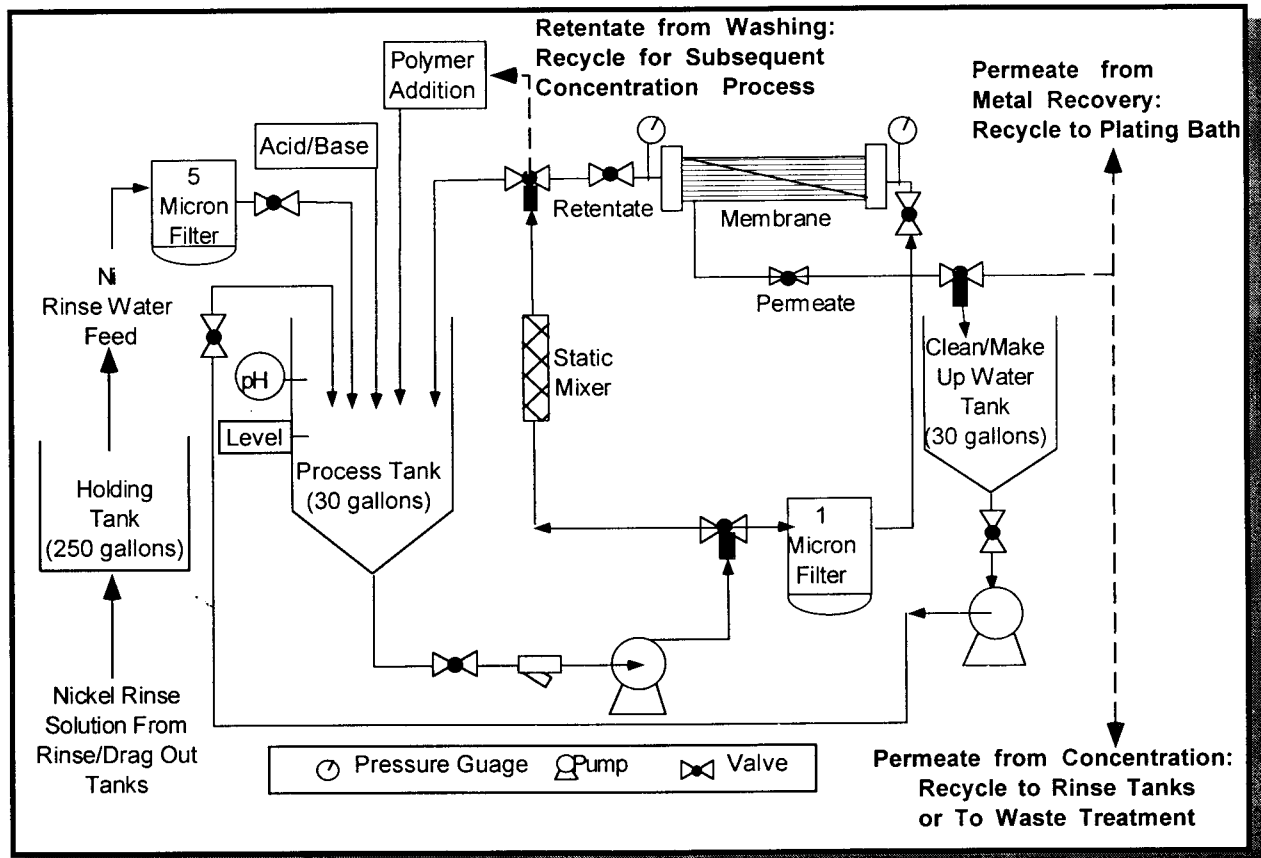


FIGURE 6 - SILVEX PF TREATMENT PROCESS FLOW DIAGRAM

The PF process is performed in three phases: the binding phase, the concentration phase, and the metal recovery phase. Each phase of the PF process uses the same equipment, as shown above in Figure 6. The flow through the system for each phase is controlled by the valves located throughout the system. For example, during the binding process the rinse solution does not flow through the ultrafiltration membrane until the pH has been adjusted and the polymer/metal solution is fully mixed. Process details of each phase are described below.



The operating sequence of the **Binding Process** is as follows:

1. Approximately 30 gallons of nickel rinse solution is transferred from the holding tank through a 5 micron filter and into the process tank and is circulated through the system, but not through the membrane.
2. While the rinse solution is circulating through the system, the polymer solution is added and the pH of the rinse solution is adjusted, by acid/base addition, for binding.
3. The static mixer is turned on to mix the polymer with the rinse solution. The mixed solution is referred to as the "retentate" solution. The retentate solution flows from the process tank, and is pumped through the static mixer and back into the process tank.
4. After the pH is adjusted and the polymer solution is fully mixed with the rinse solution, the direction of the process valves is changed so that the retentate solution flows through a 1 micron filter and then across the membrane and returned to the process tank. This process is called "total recycle mode".
5. Inlet and outlet pressures for the membrane are monitored and allowed to stabilize. Upon stabilization of the trans-membrane pressure, the valve for the permeate port is opened to discharge the permeate water. The system is operated in total recycle mode, with both permeate and retentate solutions being returned to the process tank, to allow for maximum binding of the nickel to the polymer.

The operating sequence of the **Concentration Process** is as follows:

1. After the total recycle mode, the direction of the permeate line valve (valve below membrane) is changed so that the permeate water is collected in the clean water tank, and the concentration process begins.
2. The concentration process continues by recirculating the retentate solution across the membrane, and collecting the permeate water during each pass across the membrane.
3. The solution is concentrated down to a minimum volume, subsequent portions of rinse water are transferred from the nickel rinse tanks, and the process continues until all portions are treated.

The operating sequence of the **Metal Recovery Process** is as follows:

1. Upon completion of the concentration process, the retentate solution is recirculated through the system for pH adjustment in the same manner as in the binding process. In the metal recovery phase, the pH of the concentrated polymer/metal solution is adjusted to separate metal from the chelating polymer.
2. After separation, the process of diafiltration is performed by pumping the solution through the ultrafiltration membrane.
3. Water is introduced from the make up water tank at the same rate as permeation.
4. The reclaimed nickel solution is collected during this process. On average during the Silvex demonstration, 90% of the original nickel that entered the system during the concentration phase is reclaimed.



Polymer Washing/Regeneration Process: The chelated polymer is collected during the metal recovery process. The regenerated polymer recovered during the metal recovery phase is then washed with a minimal volume of water and is returned to the feed tank for subsequent runs.

Reuse/Recycling of Stream Components:

- At the end of the polymer washing process, the polymer solution is transferred back to the polymer holding tank and is used in the next concentration process to treat another batch of rinse water.
- The permeate from the metal recovery process consists essentially of nickel chloride/nickel sulfate and is available to be recycled back to the original plating bath to replenish evaporated solution.
- The permeate from the concentration process contains nickel concentrations ranging from 0.1-1.0 ppm, and is available for recycling to the rinse tanks or discharge to the sewer.

Thus, this process recycles the metal ions in the waste water without producing a hydroxide sludge, which is normally landfilled, and enables water reuse. Also, the PF treatment agent (the polymeric material) is reused in the process, conserving additional resources.



P2 Technology Performance

This section will present performance data for the PolyIonix PF technology as a result of its application at Silvex, in Westbrook, ME. This section gives the PolyIonix PF technology performance goals in this application. The technology's performance in the selected application is described by summarizing the application runs made and the performance achieved.

P2 Performance Goals

The primary goals of the PF technology implementation at Silvex were to evaluate equipment design, PF process variation, and strength of PF technology in an industrial setting. The following presents additional goals of the PF technology implementation at Silvex.

ADDITIONAL OBJECTIVES:

- ▶ Verify the PF technology's effectiveness by achieving less than the discharge limit of 2.37 ppm nickel in the permeate from a feed concentration of approximately 100 ppm nickel.
- ▶ Operate the PF system continuously for 4 weeks treating 90 gallons per day.
- ▶ Verify return of purified metal ions to plating bath with no detrimental effect on plating operations. Perform Hull-Cell tests using the reclaimed nickel solution from the metal release processing to evaluate the chemical similarity and plating capability of the treated nickel solution to the original plating bath.
- ▶ Run split batch (mini-batch) process (as described on pages 11 and 13) to obtain field data for comparison with laboratory test results.
- ▶ Recycle water back to the process.
- ▶ Demonstrate sludge reduction at Silvex through operation of the PF technology for two of the three nickel plating lines at Silvex.
- ▶ Demonstrate recycle of nickel metal.

P2 Technology Application Test Cases

This section presents the tests run at Silvex to accomplish the goals presented in the previous section. All tests referred to in this section apply to the recent PF technology application at Silvex. The results of these test cases are presented in the next section.



Run split batch processes: The purpose of running split batch processes was to compare the results obtained from the field application to previous results obtained in the laboratory.

- Rinse solution was transferred from the nickel rinse tanks and/or the drag out tank to the holding tank. Approximately 90 gallons of rinse solution per process was transferred from the nickel rinse tanks to the holding tank. The concentration of nickel was determined using an atomic absorption (AA) instrument.
- As the process tank in the PF system at Silvex can treat approximately 30 gallons of the nickel rinse solution per batch, the 90 gallon process volume was split into three batches of 30 gallons each. Polymer solution was used to treat each batch of the rinse solution.
- Each batch of treated nickel rinse solution was subsequently concentrated using the PF technology, with subsequent batches added to the concentrated retentate from previous batches until the entire volume of rinse solution was concentrated to approximately 3500-4500 ppm. The retentate solution was then recirculated through the system, and the metal release process was performed. Polymer washing was performed using the permeate water or de-ionized water.

Perform Hull-Cell tests: To demonstrate that returning the reclaimed metal to the plating solution is acceptable, an evaluation (in this case, Hull Cell testing) is required to compare the chemical similarity and plating capability of the reclaimed metal solution with the original plating bath. The following procedure was followed:

- A sample was collected from the reclaimed nickel solution generated from the metal release process.
- The amount of the sample required to run the Hull Cell testing was determined using the ratio of the nickel concentration in the reclaimed solution sample to the plating bath.
- The correct amount of sample and 1000 ml of the plating bath solution were transferred to the Hull Cell plating tank.
- A metal coupon was submersed in the Hull Cell tank containing mixed reclaimed metal and plating solutions to plate nickel on the coupon. As a control, nickel was plated on another metal coupon using the plating bath solution without addition of reclaimed metal. After each of the metal coupons was plated, they were compared.

The results of the PF runs and the Hull-Cell tests are presented in the following section.

P2 Technology Application Results

This section presents the results of the Silvex application of the PF technology. The results presented herein address each of the performance goals as previously stated, and each of the application test cases previously described. In general, the performance goals were achieved, with few exceptions. The data presented represent a typical day of processing at Silvex.

Run Split Batch Process: Figure 7 presents the concentration process results from a normal day of processing. Tests were conducted in two phases: the concentration phase and the metal recovery phase. An initial volume of 180 gallons of nickel rinse water, with a nickel concentration of 106



ppm, was processed in approximately 6 hours. The rinse water was treated using the split-batch process in 6 batches, with sample collection during each batch. The nickel concentration in the final retentate of this run was approximately 3500 ppm. The nickel concentration in the permeate (maximum 0.6 ppm) was well within the daily discharge limit of 2.37 ppm. Table 2 presents the concentration data for the nickel rinse solution processed on 22 September 1997.

CONCENTRATION PROCESS RESULTS:

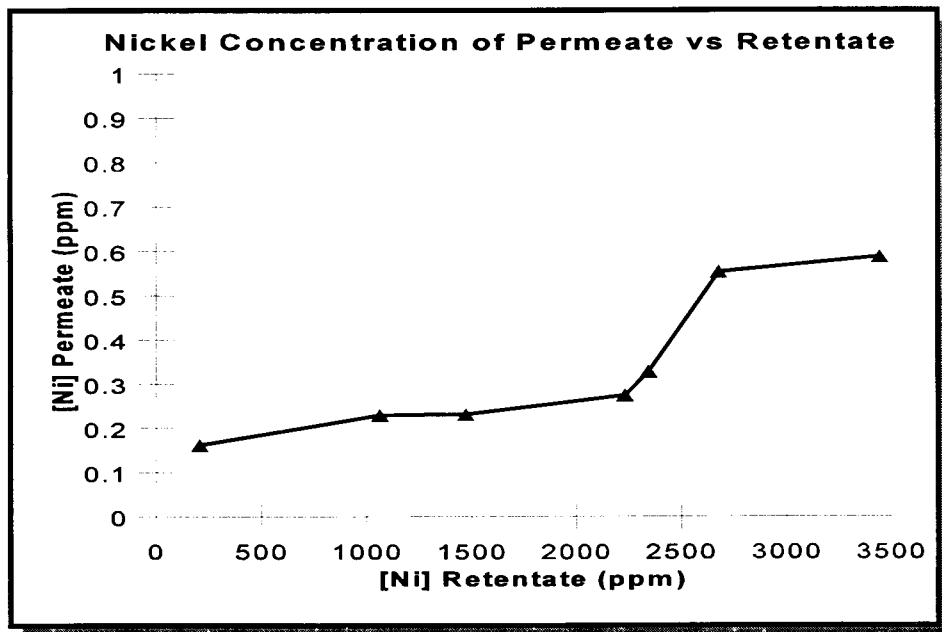


FIGURE 7 - NICKEL CONCENTRATION OF PERMEATE VS RETENTATE

TABLE 2 - CONCENTRATION PROCESS DATA - 22 SEPTEMBER 1997

[Ni] Retentate (ppm)	210	1100	1500	2200	2300	2700	3500
[Ni] Permeate (ppm)	0.2	0.2	0.2	0.3	0.3	0.6	0.6

As presented in Figure 7 and Table 2 above, the split-batch PF technology processed 180 gallons in a day and was effective at reducing the nickel concentration in the effluent to well below 1 ppm.

Figure 8 presents the results of the metal recovery phase. Using approximately 3-4 volume equivalents of water, 78% of nickel was recovered in this phase. Table 3 presents the metal recovery data for the nickel rinse solution processed on 22 September 1997.



DIAFILTRATION (METAL RECOVERY) PROCESS RESULTS:

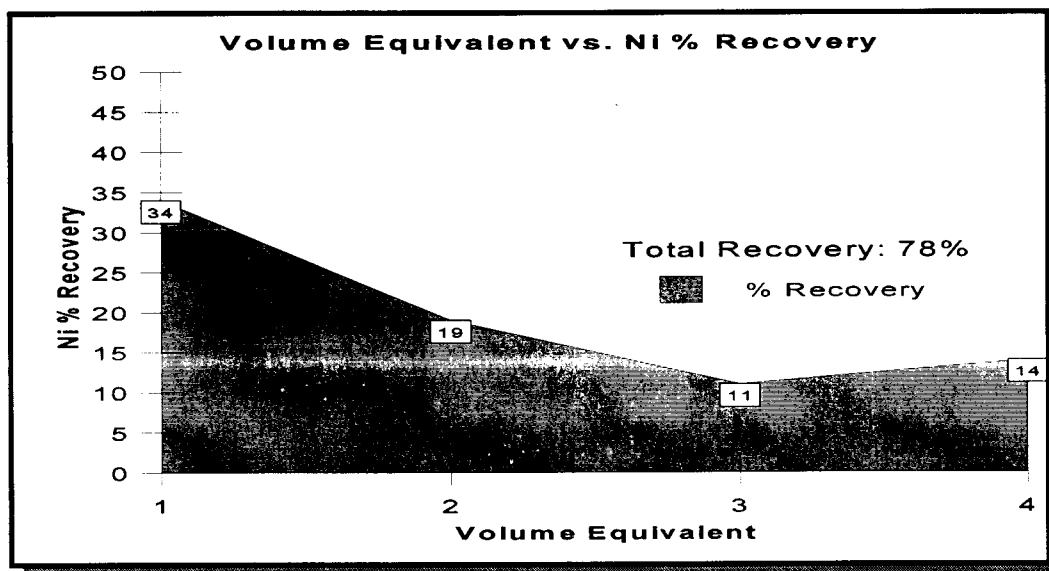


FIGURE 8 - % RECOVERY NICKEL

TABLE 3 - METAL RECOVERY PROCESS DATA - 22 SEPTEMBER 1997

Volume Equivalent	Ni Conc. (ppm)	Amount of Ni (g)	% Recovery
1	2468	24.68	34
2	1348	13.48	19
3	826	8.26	11
4	996	9.96	14
Total		56.38	78

Perform Hull Cell tests: Hull Cell testing was performed as described in the previous section. The Hull Cell test is a qualitative measurement used to demonstrate no adverse affect on the plating operation by using the final nickel solution in the plating process. After the coupons were plated, they were compared. The following presents the results of this comparison.

- The first Hull Cell test result showed that the coupon plated in the reclaimed metal solution had a dark streak mark on the right corner, as compared to the metal coupon plated in the plating bath solution. This streaking disappeared when the pH was raised.
- After pH adjustment, the plating on the Hull Cell coupon appeared to be of the same quality as the plating bath coupon demonstrating that the collected nickel solution can be recycled to the plating solution without further adjustments to the nickel solution.
- The Hull Cell tests indicate that recycling the reclaimed metals into the plating bath does not affect the composition of the plating solution and does not compromise the plating composition or process.

Wash polymer solution with permeate water: The collected permeate water was used to wash the polymer solution for regeneration in 90% of the operations at Silvex as described in the previous section. The following conclusions are drawn from this study:

- The use of permeate water to wash the polymer is not detrimental to the PF process.
- Deionized (DI) water was used twice for polymer washing to verify the lack of difference between using DI water and permeate water.
- The permeate water from the concentration process could be used for metal releasing, thereby reducing usage of DI water and resulting in complete recycling of the water.

Performance Compared to Existing/Traditional Technology

This section provides a comparison of traditional technology and the PolyIonix PF technology. Table 4 compares the PF technology with other technologies commonly implemented to achieve metal removal from industrial waste water. As presented in Table 4, the PF process generates no secondary waste (air emissions, additional waste water, or sludge).

TABLE 4 - COMPARISON WITH OTHER TECHNOLOGIES*

Process	Metal Recyclability	Secondary Waste	Operating Expenses
Polymer Filtration	Yes	None	Low
Ion Exchange	Yes	Resin By-Product	Moderate
Evaporation	Yes	Air Emissions	High
Reverse Osmosis	Yes	Reject Solution	High
Electrodialysis	Yes	Reject Solution	High
Precipitation	No	Sludge	High

* Comparison made by PolyIonix



Cost Information

This section presents cost information associated with the design, construction, startup, and operation of the PolyIonix PF technology at Silvex. The costs estimated are in current US dollars (January 1998).

Capital Costs

Table 5 presents capital costs and other installation parameters for the PF technology as demonstrated at Silvex, which was a manually controlled unit, and projected capital costs which would be associated with a fully automated commercial unit of the PF technology. The commercial PF unit is based on the same capacity (1-1.5 gpm) as the Silvex unit; however, it will incorporate automation such that the unit will fit into the existing process line at the end facility.

TABLE 5 - CAPITAL COST SUMMARY *

	Actuals Silvex	Fully Automated Commercial Unit
Mobile Unit Construction (1-1.5 gpm) (Reaction Reservoir, Pumps, Plumbing, Controls, Chelating Polymer, Ultrafiltration Membrane Cartridges, Acid and Base Tanks)	\$25,000	\$40,000-50,000; or lease arrangement
Installation Time	2 hours	2 hours
Plant Downtime	0	0
Infrastructure Change Requirements	move 1 pump; add holding tank	provide additional piping to fit existing process.

* - Projections made by PolyIonix, based on 1.5 gpm flow.

Operating Costs

This section presents the cost parameters for the Silvex PF technology application. In addition, the cost parameters and annualized operating cost projections for a fully automated commercial unit are presented in Table 6.



TABLE 6 - OPERATING COST SUMMARY FOR THE PF TECHNOLOGY

	Silvex	Fully Automated Commercial Electroplating Unit	
	Operating Parameter	Operating Parameter	\$/yr
Labor	8 hrs/day for 16 days of operation ¹	2 hrs/day	7200
Utilities (electricity, water)	\$15 for 21 days of operation	\$1.50/day	500
Laboratory (AA, Hull Cell testing)	6 AA/day ² 2 Hull Cell/16 days	2-4 AA/day (do not need real-time results according to vendor)	approx. 8700 to 17300
Training	None	3 hours per operator and supervisor ³	300
Maintenance	None	\$100/month for service contract	1200
Chemicals (acid, base)	No additional ⁴	3-4 liters per cycle	1000
Monitoring	1 operators @ 8hrs/ day for 16 days of operation	Occasional operator interface, primarily for analyses	
Sewer	No incremental expense	Reduce contaminant discharge to sewer/POTW.	
Total			18,900 to 27,500

- Notes:
1. Labor provided by PolyIonix for Silvex Demonstration
 2. Atomic Absorption (AA) is performed to determine the amount of nickel in the sample. AA is a technique for analyzing metals using an element specific lamp that emits a characteristic light spectrum. A sample is heated in a flame or graphite furnace and the light beam is passed through it. When the sample absorbs light, an energy loss is detected and is translated to a concentration of a specific metal in the sample. The technique detects one metal at a time. Hull Cell testing is performed to study the electrodeposits formed from various electrolytes.
 3. No additional training costs incurred associated with this demonstration as PolyIonix provided personnel to operate PF technology. However, training and license requirements for supervisors and operators is required for operation of the system. PolyIonix is currently generating a training manual to be used in conjunction with the PF technology.
 4. Necessary materials (acid and base for pH adjustment) were available at Silvex and were provided on an as-needed basis for the field demonstration.

Cost Comparison

A comparison of the relative costs associated with the use of traditional chemical precipitation technology at Silvex versus the PolyIonix PF technology at Silvex is presented below in Table 7. As shown in Table 7, the PF technology is competitive compared to existing technology. The comparison data was supplied by PolyIonix.

TABLE 7 - SILVEX YEARLY COST OF PF TECHNOLOGY VS CHEMICAL PRECIPITATION (¢/GALLON TREATED)

	Water	Sewer	Sludge	Treatment Chemicals	Total
PF Silvex (1997)¹	0.1	0.5	1.1 ² / (1.6) ³	2.3	4.0
Chemical Precipitation Silvex	0.3	0.5	3.2	5	9.0

1 - Based on 500 gal/day, 240 day/year.

2 - Recovered metal not reused at Silvex but sent to waste treatment.

3 - Assumes no sludge; metal value reclaimed.



Table 8 presents a comparison of capital costs, annual operating costs, and cost savings from material recovery for the PF technology versus other traditional technologies. The cost estimates were developed in a study performed by PolyIonix in November 1997.

TABLE 8 - PF VS TRADITIONAL TECHNOLOGY COST COMPARISON¹

	Capital Cost (\$)	Annual Operating Cost (\$)	Savings from Material Recovery (\$)
Polymer Filtration	45,000	18,900 - 27,500	23,000
Ion Exchange	50,000	22,000	23,000
Evaporation	36,000	162,000	3,000
Hydroxide Precipitation	95,000	35,000	0

Notes: 1-comparison made by PolyIonix

2-Flow Rate: 3 gpm

3-Water Cost: \$0.001/gal

4-Sewer Cost: \$0.002/gal

5-Electricity Cost: \$0.06/kWh

6-Metal: Nickel, 660 ppm

7-Operation: 24 hours/day for 240 days/year

8-Sludge Disposal: \$180/ton

As presented in Table 8 above, the PF technology compares favorably with traditional technologies.

Cost Benchmarks

This section provides information regarding cost benchmarks that illustrate P2 benefits derived from the Silvex PF technology application.

COST BENCHMARKS

- 2 year anticipated pay back period.
- 25 % reduction in disposal costs.
- 15 % reduction in sewer fees.
- 25 % reduction in waste transportation/treatment cost.
- 20 % reduction in sludge volume produced from nickel process.
- 5 % reduction in nickel purchase.
- Positive cash flow in the first year



Regulatory/Safety Requirements

This section provides information regarding the regulatory requirements related to the implementation of the PolyIonix PF technology system.

Applicable Regulations

This section presents regulations that apply to the implementation of the PolyIonix PF technology. Table 9 presents a list which includes, but is not limited to, applicable regulations and the regulatory authority responsible for its administration. Additional state or local regulations may apply.

TABLE 9 - APPLICABLE REGULATIONS

Applicable Regulation	Responsible Regulatory Authority
Clean Water Act	US EPA and specific state environmental agency, state, local
Occupational Safety and Health Administration	US Department of Labor, state
Resource Conservation and Recovery Act	US EPA, state
Toxic Substances Control Act	US EPA, state
Emergency Preparedness and Community Right-To-Know Act	US EPA, state

Permit Issues

No new permits were required for the PF technology application at Silvex. As the PF technology is integral to the process stream and both components - purified metal ions and final effluent - can be returned to the process line, no additional permits are required for the system. For the application at Silvex, most of the final effluent was sent to the existing on-site waste treatment system, but some of the final permeate was recycled back into the process. The PF technology mitigates potential impacts to air and water, as well as hazardous waste related issues, as described below.

- **Hazardous waste management** - The PF technology produces no additional sludge, toxic or inert, which needs to be landfilled. When incorporated into a commercial electroplating installation, the volume of sludge from the electroplating process will be reduced, which in turn, reduces the landfill disposal costs for the facility.
- **Water** - The levels of contaminants in the effluent from the PF technology are below the regulatory discharge requirements, reducing the quantity of metal contaminants discharged to the sewer or POTW. Risk of permit violations is lessened.
- **Air** - As both components of the effluent are returned to the process line, and no fugitive air emissions exist, no air permits are required. While air emissions are not generated by the PF technology, PolyIonix will be monitoring worker exposure in future field trials.



Regulatory Interaction

This section identifies waiting times required for permits and formal approvals required from regulators required for the implementation of the PolyIonix PF technology and other regulatory interaction which has occurred regarding the PF technology.

For the Silvex application, there were no permitting issues which caused delays in project execution resulting from regulatory review and approval of plans.

PolyIonix has been a participant in the Research & Technology Work Group's Approaching Zero Discharge (AZD) Program, which falls under the Metal Finishing Sector of the EPA Common Sense Initiative (CSI) Program, for the past 18 months. The PF system was selected by CSI's Peer Review Group as the demonstration technology for the AZD Program. PolyIonix will be working with the EPA and the CSI program administrator, Camp, Inc. of Cleveland, OH, to demonstrate the AZD capabilities of the PF technology in several metal finishing facilities.

Health/Safety Issues

This section discusses health and safety issues associated with the PF technology application at Silvex:

Sulfuric acid is used in the pH adjustment. Material Safety Data Sheets should be available for workers in contact with the system. Although the pH adjustment will be automated in the commercial unit, operator awareness and safe work practices are necessary while operating the system.

The polymer is inert and therefore presents no health hazards to operators or technicians.

A small leak of a couple of gallons occurred during the piping modification and pump changeout at Silvex. The release was promptly contained and cleaned up. No health and safety problems occurred as a result of the leak.

Operator safety training is required for the PF technology. PolyIonix is currently developing an operator training manual and will provide appropriate training to on-site personnel on an application-specific basis.

The PF unit incorporates operator and system safety features such as level controls, pressure sensors, and temperature sensors.

Exposure to contaminated water and metal contaminants is decreased as the PF effluent meets POTW water discharge limits.



Lessons Learned/Implementation Issues

The lessons and issues presented below are based on the PolyIonix PF technology application at Silvex in Westbrook, ME. Lessons learned in both design and operations areas were derived from information provided by PolyIonix and Silvex.

Design Issues

Lessons learned regarding issues related to the design of the PF unit and technology include:

The following are lessons learned during development of the technology which have already been incorporated into the design of the standard unit:

- ▶ Modify design to add the capability to by-pass the membrane for recirculation to aid in mixing, and to increase the residence time.
- ▶ Fouling of the ultrafiltration membranes can be minimized by the removal of suspended solids with the use of prefilters (5 um), and the reverse flow design which was used at Silvex.
- ▶ Adequate trans-membrane pressure (TMP) is required to maintain proper flow. A minimum TMP of 10 psi is necessary to accomplish this.
- ▶ Install valve between in-line filter and recirculation line to keep retentate solution from backing up to the filter line.

The following are lessons learned during development of the technology which need to be accounted for in tailoring the unit to a specific application setting:

- ▶ Automation of equipment operation is desirable to reduce labor requirements and to ensure proper operation of the PF technology.

The following are lessons learned during development of the technology which have been addressed through further development since completion of the Silvex demonstration:

- ▶ Self-containment in the PF unit is required to contain small leaks or spills which may occur during routine maintenance procedures.
- ▶ System drains are needed at the following locations within the PF system:
 - (1) Main system drain located at the main low point of the system,
 - (2) Ultrafiltration membrane changeout drain located near the cartridges,
 - (3) Manifold drain.

The drains will be user controlled such that they will drain into a bucket or other container and will only do so when manually controlled by the user.

Implementation Considerations

Based on experience with the PolyIonix PF technology application, lessons learned relative to future implementation of the technology include:



The following are lessons learned during development of the technology which have already been incorporated into the design of the standard unit:

- ▶ Include proper and adequate pre-filtration to remove particles greater than 5 μm in size.
- ▶ Proper waste stream characterization is mandatory to assess the applicability of the PF technology on the waste stream. Waste stream characterization aids in determination of appropriate polymer, and the necessity for additional pre-filters or pretreatment.
- ▶ Sample ports (spigots or valves) should be added to the system on the permeate and retentate lines to ease collection of samples from the system.

The following are lessons learned during development of the technology which need to be accounted for in tailoring the unit to an application setting:

- ▶ A local service requirement exists, which includes physical inspection of the system by PolyIonix and monitoring of the polymer level performed on a monthly basis.
- ▶ Purified metal concentrate can be returned to the plating bath with no detrimental results.
- ▶ PF permeate compares favorably with typical city water, thus reducing water usage without increasing the load on the existing deionization systems.

Benefits Derived From Application

This section presents a list of issues identified by PolyIonix and the PF technology user as benefits as a result of installation and implementation of this technology at Silvex.

- ▶ Return approximately 85% of water (by volume) back into process. This reduces both water purchase and POTW discharge costs.
- ▶ Return purified metal solution back into process.
- ▶ No hydroxide sludge is produced, eliminating a typical waste stream.
- ▶ No landfill disposal is needed, thus creating regulatory and economic benefits.
- ▶ Polymeric material is reused in the process, thus conserving resources and avoiding a secondary waste stream. The polymer is returned to the holding tank and run through the system again.
- ▶ Purified metal ions can be returned to the plating bath for reuse without loss of plating performance.
- ▶ The PF technology showed metal removal to levels of less than 1 ppm, which is well below regulatory discharge limits of 2.37 ppm for this location.
- ▶ The PF system can reduce the amount of nickel required for purchase by approximately 5%.



Limitations In Application

This section presents key issues that were identified by PolyIonix as limitations of PF technology as a result of installation and implementation of this technology at Silvex.

LIMITATIONS

- ▶ Must have a well defined waste stream in order to properly assess treatment of the target metals.
- ▶ Process (specific polymer used, process conditions, etc.) must be designed for each waste stream type. Care must be taken in design to assure specificity.
- ▶ Best performance at low concentrations of contamination (less than 500 ppm Ni as determined through laboratory and field experience).
- ▶ The degree of automation required may vary depending on customer requirements.
- ▶ Ultrafiltration membranes can foul from oil and other gross contaminants. With sound operating and cleaning procedures, and the reverse process flow design, membrane life-cycle is considered to be at least one year.
- ▶ Some chelations and metals, such as chromium, compete with the target metal, nickel, for polymer binding sites. Current practices and procedures permit binding but not release, and therefore cannot process the chromium.
- ▶ Equipment design is dependent on process scale.
- ▶ PF technology has not yet been utilized in larger-scale implementation, therefore scale-up has not been proven. Scale-up is dependent on process, flow, time, degree of automation, and volume requirements. System components potentially effected are the pipe diameter, pump capacity, and number and size of membranes required.
- ▶ The useful lifetime of the polymer has not yet been determined, as failure has not yet occurred. Additionally, disposal procedures of any spent polymer which may result have not been determined. These issues are currently being addressed.

